A MASS SPECTROMETER FOR THE STUDY OF SURFACE IONIZATION

BY
MORTON BATLAN EPSTEIN
B.S., New York University, 1937

AN ABSTRACT OF A THESIS

SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS
FOR THE DEGREE OF DOCTOR OF PHILOSOPHY IN
CHEMISTRY IN THE GRADUATE SCHOOL OF
THE UNIVERSITY OF ILLINOIS, 1942

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Introduction

In 1923 while studying the thermionic properties of tungsten filaments coated with caesium, Langmuir and Kingdon[1] established the existence of surface ionization. This led to numerous studies[2-7] of the ionization of alkali metals on tungsten, tantalum, molybdenum, and platinum. Rodebush and Henry[8] observed that alkali halides underwent surface ionization on oxygen coated tungsten. Phipps, Hendricks, and Copley[9] using molecular beams in place of saturated vapors measured the ionization of potassium halides on tungsten and oxygen coated tungsten. Johnson and Phipps[10] introduced a differential method for measuring the currents and extended the measurements to the ionization of sodium halides on tungsten.

The present investigation undertook to build a mass spectrometer suitable for studying surface ionization. Such an instrument would permit a more detailed study of the ionization of salts and would extend the range of studies to negative ions and electron affinities of the halogens. Studies[1] of negative ions which have been made used the saturated vapor method and were inconclusive. The instrument was of the Bleakney[12] type and used crossed magnetic and electric fields. This design, unlike that of other instruments, focusses all ions of the same e/m regardless of initial velocity and direction. This thesis describes a continuation of the work begun by H. M. Clark.[13]

Theoretical

The force acting on a charged particle in an electrical and magnetic field is

$$F = m\, dv/dt = e(E + v \times H).$$

The fields are homogeneous and orthogonal; applying the

linear transformation, $v = u + V$, where

$$u = E \times H/H^2$$

we obtain

$$m\, dV/dt = e(E + u \times H + V \times H),$$

which may be reduced by standard vector formulae to

$$m\, dV/dt = -e(V \times H).$$

The paths are therefore a combination of a uniform linear motion and a uniform circular motion and are cycloids. The focal distance is equal to the circumference of the generating circle and is $2\pi nmE/eH^2$. Resolution of the instrument is limited by the dip of the curtate and the return loop of the prolate path. The widths of the dip and loop were calculated for the Maxwellian root mean square velocity by Clark.[13] In general, it was observed that the magnetic intensity, the path length and the mass of the ion were more important than the temperature in determining the resolution of the instrument.

Experimental

A permanent magnet was used to avoid fluctuations in the magnetic field. The casting was of "Alnico" in order to obtain a high magnetic intensity. Its shape was that of a pair of "Dees," the pole-faces being connected by two heavy yokes. The pole faces were 1¼" by 3" and 1.52 cm. apart.

The magnet was suspended from a counter-balanced sliding mechanism by means of three ¼" Monel metal tubes attached to the magnet by compression fittings. Monel was chosen to reduce liquid air consumption. The counter-balanced sliding mechanism was constructed of two pairs of steel ball bearings which slid on either side of two vertical hardened steel rods and could be clamped at any position.

The electric field was provided by applying a potential across two parallel pyrex plates coated with platinum. The platinum was applied by vaporization from a filament in vacuo. The plates were 0.884 cm. by 0.51 cm. and were 1.05 cm. apart.

An ionizing filament and a pair of collector filaments, one for positive ions and one for negative ions, were supported

by small pyrex posts sealed to one of the pyrex plates. The collector filaments were spaced 1.9 cm. on either side of the centrally-located ionizing filament. The filaments were 0.140 cm. above the lower plate.

A molecular beam of potassium chloride was furnished by a quartz furnace heated by a tungsten coil. The beam entered the electric field through a round hole 0.33 mm. in diameter in one quartz plate and struck the ionizing filament. A round hole 0.58 mm. in diameter in the lower plate permitted unionized molecules to leave the field.

The plates, filaments, furnace, and shields were contained in a flat glass cell 90 by 100 by 11 mm. inside dimensions which could just slip between the pole-faces. Leads to the parts in the cell emerged from the vacuum through tungsten-to-pyrex seals.

Two mercury vapor diffusion pumps supported by a Cenco Hyvac pump evacuated the cell. Liquid air traps were placed before the vapor pumps and before the cell. The line included a 5-liter stabilizing flask, a McLeod gauge and a magnetically-operated cut-off which exposed no iron to the vacuum.

The ion currents were amplified by means of a vacuum tube amplifier based on the FP-54 pliotron and measured by a high sensitivity galvanometer. The over-all sensitivity was 55,000 mm. per volt and currents of 10^{-15} amperes could be detected.

Results and Conclusions

With KCl as the salt, currents of from 10^{-10} to 10^{-12} amperes were found. Positive and negative ions in about equal numbers were observed. No peaks, however, could be separated from the background currents and the ions could not be identified. This difficulty was traced to non-homogeneous electrical and magnetic fields. Computations of field strength based upon the method of conformal transformations indicated that the electrical field was highly non-homogeneous.

Calculations show that a magnet having poles faces 8 by 15 cm. and 3 cm. apart would provide adequate resolu-

tion. An alternative design based upon Dempster's field arrangement was suggested.

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VITA

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